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To cite this article: A Stolbovsky *et al* 2020 *IOP Conf. Ser.: Mater. Sci. Eng.* **969** 012087

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The Structure of Annealed Hafnium Bronze Subjected to Severe Plastic Deformation by High Pressure Torsion

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Abstract. The structure and mechanical properties of low-alloyed hafnium bronze (Cu-0.78 wt. % Hf) under severe plastic deformation by high-pressure torsion (HPT) by 1, 3 and 5 revolutions of anvils at room temperature have been studied by electron microscopy and microhardness measurements. In the initial annealed state Hf is practically completely bonded in intermetallic compounds. The structure of bronze specimens deformed by HPT is stable and does not undergo any changes after unloading and long room temperature ageing. Under the deformation a dispersed submicrocrystalline structure gradient along specimen radius is formed. Average crystallite size in the radius middle is 200 nm after 1 revolution and 120 nm after 5 revolutions of anvils. It is demonstrated that in all the specimens studied including those deformed by 5 revolutions the structure is not uniform which is confirmed by microhardness measurements. Such behavior of hafnium bronze with Hf bonded in precipitates has much in common with the behavior of pure copper. However, the room temperature stability of the structures obtained by HPT demonstrates similar behavior with that of low-alloyed tin bronze.

1. Introduction

Pure copper has always been one of the most widely used materials for electrical engineering as it combines high electro- and heat-conductivity with good plasticity. Particularly, copper is practically indispensable for details of contact groups in electro-technical articles or heat exchangers operating under enhanced loads. However, its application as a constructional material is limited by its not high enough strength and relatively low melting temperature.

Constant search of possibilities to enhance mechanical characteristics of copper for its wider application as a constructional material with unique properties has been carried on for several last decades [1]. And only after an appearance of a new approach to creation of materials with high density of internal defects [1–15], copper was suggested as a valuable constructional material. However, copper articles have still certain principal drawbacks such as low thermal stability of the structure [10, 12–15].

Impurities and doping elements are known to increase the recrystallization temperature of copper even in case of their very low concentration. Doping of copper with elements forming solid solutions with high concentration, such as, for example, tin, is not effective for an enhancement of thermal stability of the structure [16]. It is believed [17–26], that doping of copper with elements demonstrating the lowest solubility in it (Zr, Hf, etc.) enables to achieve the highest temperature of the beginning of



recrystallization. At the same time, their concentration increasing upper 0.05 % not always results in noticeable increase of recrystallization temperature [17, 18].

The goal of the present work was to study hafnium bronze with minimal content of the doping element in solid solution subjected to severe plastic deformation by HPT in order to determine the structure formed and the level of strength characteristics.

2. Materials and methods

The composition of hafnium bronze used in the present study is given in Table 1.

Table 1. Chemical composition of hafnium bronze, wt. %

Element	Hf	O	Fe	Ni	Zn	Sn
Content, wt. %	0.78~0.28	~0.001	~0.005	~0.005	~0.005	~0.005

The hafnium bronze after melting was subjected to hot forging at 600°C. After that it was mechanically treated and annealed for homogenization at 800°C, 1 h with following cooling in a tubular vacuum furnace enabling to carry out annealing at the pressure of 10^{-2} – 10^{-3} Pa.

Deformation was carried out in open Bridgeman anvils the diameter of 10 mm at room temperature, by 1, 3 and 5 revolutions of anvils, at the pressure of 6 GPa and angle speed of 0.3 revs/min.

The structure of as-deformed and annealed specimens was studied by transmission electron microscopy in Philips-CM30 SuperTwin and JEM-200CX electron microscopes.

Microhardness after the HPT was measured in a special unit to Neophot-21 optical microscope, at a load of 40 g, by the techniques described in [27].

3. Results and discussion

In the initial annealed state the bronze had polycrystalline structure with crystallite sizes of 20–40 μm , that is, much smaller than in case of pure copper and tin bronze [16]. Besides, there were second phase particles in the structure, the sizes of 1–4 μm , which is in agreement with the results obtained in [18], where hafnium bronze with 0.9 wt. % Hf was studied and in the initial annealed state it contained Cu_5Hf particles the sizes of ~ 3 μm . Thus, according to the phase diagram one could expect that after such treatment the main part of hafnium would be bonded in Cu_5Hf intermetallic compound. Microhardness in the initial annealed state was 760 ± 20 MPa, which is very close to its values for polycrystalline copper of commercial purity [16].

It should be noted that the calculated strain under the HPT depends on the distance from the rotation axis, and deformation is distributed non-uniformly along specimen radius increasing from center to periphery. According to the data of TEM studies, after the HPT the structure of hafnium bronze is gradient along disc radius; it is dispersed submicrocrystalline, with mainly high-angle boundaries between crystallites. After the deformation by 1 revolution of anvils, in the central part of the disc, where the strain is minimal and the structure is less fragmented, the average size of crystallites is about 200 nm (Figure 1 a, b). Specific contrast inside crystallites indicating high level of internal stresses is clearly seen in TEM images. Some crystallite boundaries are low-angle, resembling dislocation pile-ups, though thin and straight high-angle boundaries are also present. In some electron diffraction patterns there are closely located reflections forming radially extended aggregates in the Debye rings, but the number of such reflections is small. Such electron diffraction patterns can be considered as transitional from point-wise characteristic of coarse-grained structure to ring-wise indicating the submicrocrystalline structure formation.

With increasing distance from specimen center the strain increases, and the structure gets more dispersed, with smaller average crystallite sizes. The gradient character of microstructure is confirmed by microhardness measurements along specimen radius. Thus, after 1 revolution of anvils the microhardness is 1740 ± 60 MPa in specimen center, 1820 ± 20 MPa in the radius middle and 2050 ± 40 at the edge (Figure 2a). With strain increasing (increased number of revolutions) the microhardness

is increasing throughout the specimen, and its values are equalizing along the radius. This behavior is quite expectable, but in the case under consideration the values of microhardness are still somewhat different even after the HPT by 5 revolutions. Thus, in hafnium bronze the structure is gradient and depends on the distance from specimen center even after the highest deformation by HPT.

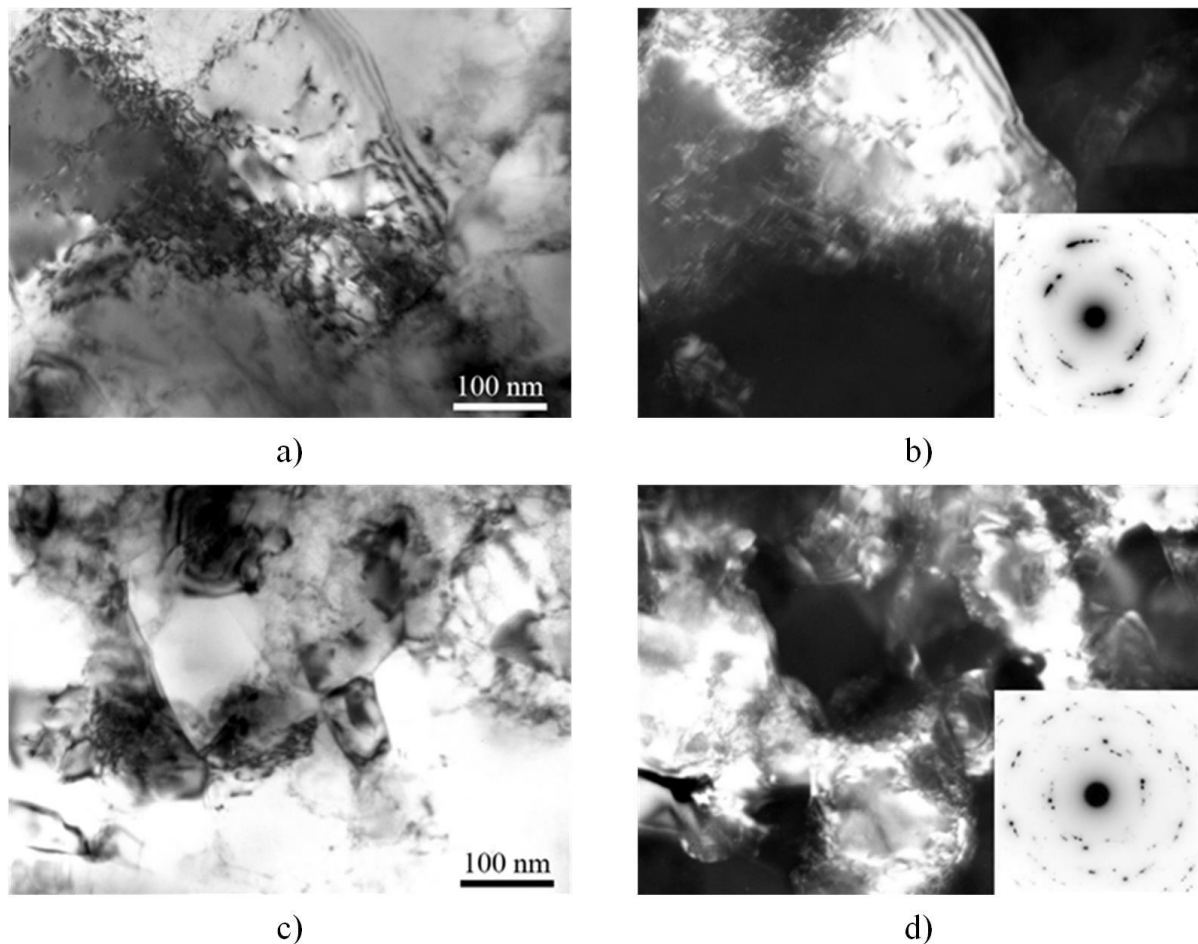


Figure 1. Electron micrographs of the structure and electron diffraction patterns in central parts of hafnium bronze specimens after 1 revolution (a, b) and 3 revolutions (c, d) of anvils: (a, c) – bright-field images, (b, d) – dark-field images with electron diffraction patterns in inserts.

Since after the HPT by 3 and 5 revolutions the microhardness is more uniform along the specimen radius than after 1 revolution, one could expect more uniform structure as well. According to TEM data, the structure is really more dispersed and uniform (Figure 1 c, d). After 3 revolutions the average size of crystallites in the radius middle is 150 nm. Crystallite boundaries are curved, and inside the crystallites there is a specific contrast characteristic of the highly stresses state, which indirectly indicates high level of internal stresses and peculiar non-equilibrium state of boundaries. However, practically in all the electron diffraction patterns the Debye rings are still not populated with the high enough number of reflections (Figure 2d).

As in the periphery of specimens after 1, 3 and 5 revolutions the microhardness increases practically up to one and the same level of 2050 ± 40 MPa (Figure 2a), one could expect similar microstructure in these areas.

The average size of crystallites in the periphery of specimens deformed by 5 revolutions is about 120 nm. However, even in this case the structure is not uniform and is very similar to that obtained after the HPT with lower deformation degree. In the electron diffraction patterns the Debye rings are not densely

populated with uniformly distributed reflections. However, in some periphery areas of discs after this treatment there are elements of structure indicating the relaxation processes. There are some grains with thin straight boundaries, crystallites of elongated shape and areas with non-uniform distribution of contrast inside crystallites. The relaxation processes directly under the deformation can limit both grain refinement and microhardness increasing in this material.

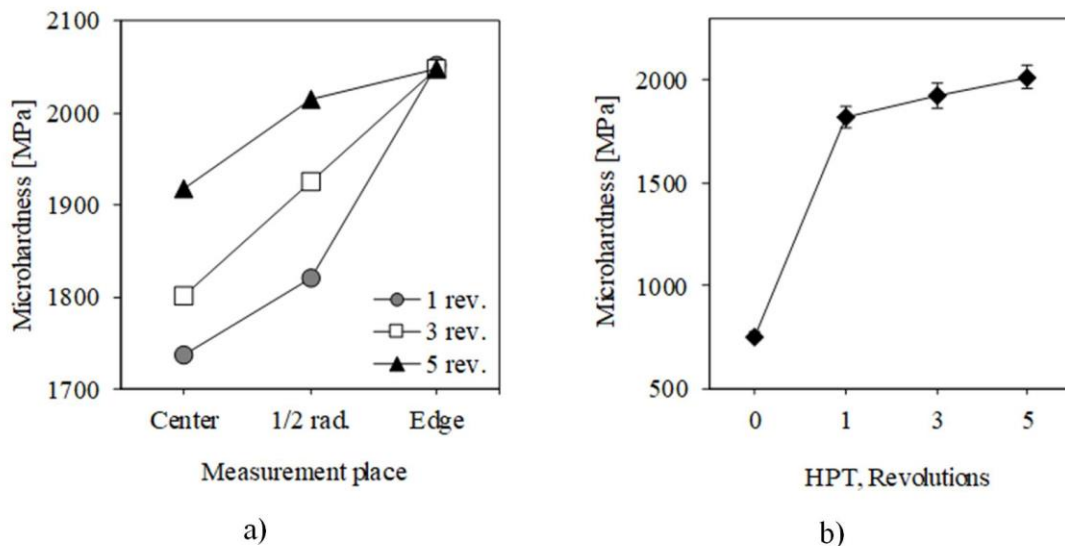


Figure 2. Microhardness along the radii of specimens deformed by HPT with different number of revolutions (a) and its dependence on the number of revolutions (in the radius middle) (b).

The strengthening of hafnium bronze with strain increasing is demonstrated by Figure 2b, in which the microhardness is shown dependently on the number of revolutions. The microhardness increases gradually, but its growth is not as pronounced as in, for example, tin bronze [28], and the values are intermediate between those obtained for the tin bronze and the commercial purity copper of M1 standard. In hafnium bronze pronounced strengthening is achieved after only 1 revolution of anvils, as in the case of tin bronze. However, with further increasing of strain some elements indicating relaxation processes appear in the structure, as it was observed in copper specimens, and the microhardness increasing is slowed down. At the same time, the structures obtained in hafnium bronze are stable at room temperature, as in tin bronze, whereas in copper specimens after unloading the structure changes at room temperature ageing.

It can be concluded that HPT even by 1 revolution enables to achieve more pronounced refinement of the structure of hafnium bronze compared to its deformation by ECAP [19,23], due to higher strain and in spite of the gradient changing of the structure along specimens radii. Besides, the structure after HPT compared to that after ECAP is more uniform in sizes, with dominating equiaxed crystallites and without pronounced deformation bands and extended twins characteristic of the ECAP-ed specimens.

4. Summary

Formation of structure and properties of hafnium bronze homogenized by annealing at 800°C and subjected to severe plastic deformation by high-pressure torsion have been studied.

It is demonstrated that HPT results in much more pronounced refinement of the structure of the hafnium bronze compared to ECAP. The structure of specimens after HPT is not only more dispersed, but also more uniform, with dominating equiaxed crystallites.

It has been established that the structure of the bronze after HPT is stable at room temperature and does not undergo any changes after unloading and prolonged ageing.

According to the data obtained, after the HPT by 1 revolution of anvils the structure of hafnium bronze is gradient along disk radius, it is dispersed and submicrocrystalline, with average crystallite size

of 200 nm (in the radius middle), with dominating high-angle misorientation between crystallites. With the strain increasing (when the distance from the discs center is increased) the structure gets more dispersed. The average size of crystallites after the HPT by 5 revolutions is about 120 nm in the periphery parts of disks. However, even after 5 revolutions the structure is still not uniform along the radius, which is confirmed by microhardness measurements, and considerable scattering in crystallite sizes is observed. These features indicate the proceeding of relaxation directly under the deformation, which can result in limitation of grain refinement and microhardness increasing under such treatment of this material.

It can be concluded that the behavior of hafnium bronze with the doping element bonded in precipitates has much in common with that of pure copper, namely, the non-uniform structure is observed even after the HPT by 5 revolutions. On the other hand, from the viewpoint of the stability of the obtained structures at room temperature, the behavior of the hafnium bronze is similar to that of low-alloyed tin bronze.

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Acknowledgments

The electron microscopy studies were carried out on the equipment of the Collective Use Center in the Testing Center of Nanotechnologies and Advanced Materials of IMP UB RAS.

The research was carried out within the State Assignment (theme “Function”, State registration number AAAA-A19-119012990095-0).